



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: Mark Mattox

Title: **METHOD TO DECREASE IRON SULFIDE  
DEPOSITS IN PIPE LINES**

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DECLARATION UNDER 37 C.F.R. § 1.132

Commissioner for Patents  
PO Box 1450  
Alexandria, Virginia 22313-1450

I, Edward J. Valente, hereby declare that:

1. I am a scientific consultant for Synergy Chemicals, Inc., the assignee of the captioned application.
2. I am a full professor of chemistry at Mississippi College. I have conducted research in the fields of organic and physical chemistry for 25 years. My research includes the study of coordination compounds of transition metals, including iron. I have published over 100 papers in these areas. My qualifications are set out in my *curriculum vitae*, which is attached hereto as APPENDIX A.
3. I have reviewed and understood the subject patent application. I have also read and understood the final Office Action dated April 1, 2003, the Advisory Action dated August 25, 2003, and cited U.S. patent No. 6,517,617 to Chartier *et al.*
4. As the experimental data set forth below, the presence of oxygen in a composition containing the components water, a soluble ammonium salt, and tetrakis(hydroxymethyl) sulfate ("THPS") would render the composition incapable of complexing iron (II) compounds such as iron sulfide. The data thus supports my expert opinion that foams containing these components and 90 – 95 vol. % (v/v), as is the case for the foams disclosed by Chartier, cannot complex iron sulfide.
5. A control composition ("the control"), which corresponds to the composition recited in the method claims at issue, was prepared by dissolving THPS (12 wt. %) and ammonium chloride (0.8 wt %) in deionized water. An identical composition ("the test") was also prepared for comparative purposes.

6. Air was sparged rapidly into the test composition (~ 500 mL/min) during 24 hours. The test composition thus approximates the foams (90 – 95 vol. % of air) that are disclosed by Chartier. Over the same 24 hour period, control composition was prevented from contacting air.

7. Two sets of experiments evaluated the effects of the presence of oxygen in the test composition. The first set of experiments identified the evolution of phosphorus-containing compounds in each composition. The second set of experiments evaluated the abilities of these compositions to complex iron (II).

8. In the first set of experiments, the control and test compositions were separately monitored by  $^{31}\text{P}\{\text{H}\}$  NMR during the 24 hour period mentioned above.

9. As Table 1 (below) shows, the control composition, at all times during this evaluation, contained three phosphorus-containing compounds in the following relative concentrations:

Table 1. Control Composition in Absence of Oxygen

Compound	Relative Concentration (%)
unreacted THPS	19.4
tris(hydroxymethyl)phosphine	75.8
trishydroxymethylphosphine oxide	4.8

10. Of these three phosphorus-containing species, only trishydroxymethylphosphine (“TRIS”) is responsible for giving rise to the complexation of iron (II) compounds, specifically when TRIS is reacted with a soluble ammonium salt such as ammonium chloride in the presence of iron (II). THPS and trishydroxymethylphosphine oxide (“TRIS oxide”), by contrast, are incapable of complexing iron (II). Thus, as shown explicitly by the second set of experiments described below, the control composition, at all times, contained the prerequisite compounds – TRIS and ammonium chloride – that are necessary for complexing iron (II) compounds such as iron sulfide. (The presence of a small amount of TRIS oxide is ascribed to the presence of trace amounts of oxygen in the deionized water that was used to prepare the control composition.)

11. The test composition, by contrast, decomposed markedly over the 24 hour period to leave exclusively the two species, namely THPS and TRIS oxide, that do not give rise to iron (II) complexation (Table 2):

Table 2: Exposure of Test Composition to Oxygen

Compound	Relative Concentrations (%) at time $t$ (h)		
	$t = 1$	$t = 3$	$t = 24$
unreacted THPS	21.6	29.5	33.3
tris(hydroxymethyl)phosphine	71.2	49.7	0.0
trishydroxymethylphosphine oxide	7.2	20.8	66.7

12. The results of the first set of experiments thus demonstrate that a composition of THPS, ammonium chloride, water, and oxygen results in a chemically much different composition than one that is not exposed to oxygen. Specifically in the case of the test composition, TRIS is completely decomposed (i.e., oxidized), thereby rendering this composition incapable of complexing iron (II) as the data below shows.

13. The second set of experiments evaluated the abilities of the control and test compositions to complex iron (II). Portions of each composition (2 mL) were combined with 1 mL of a solution of iron (II) sulfate (100 mg of iron(II)sulfate heptahydrate in 10 mL deionized water), diluted with 1 mL water, and then monitored over time by visible spectroscopy at 500 nm.

14. A composition of THPS, water, and ammonium chloride reacts with virtually any convenient source of iron (II), including iron (II) sulfide and iron (II) sulfate, to invariably give the same iron (II) complex, which exhibits an absorption maximum at 500 nm. Thus, the intensity at 500 nm correlates directly with the complexation of iron (II). (For the convenience of obtaining accurate intensity measurements, iron (II) sulfate was employed in these experiments because iron (II) sulfide is insoluble in water. Independent tests employing iron sulfide give the identical end results.)

15. The control composition, when contacted with iron (II) sulfate, gave rise to 0.8 absorbance units (au) within 20 seconds, indicating the immediate complexation of iron (II). After 4 minutes, the absorbance had not changed, thus signaling that iron (II) complexation was complete after 20 seconds.

16. By contrast, aliquots of the air-sparged test compositions, when similarly treated with iron (II) sulfate, exhibited little to no iron (II) complexation (Table 3):

Table 3. Iron (II) Complexation by Test Composition

Time (h) that Composition Was Sparged with Air (h)	Rate of Iron (II) Complexation (au) After Time <i>t</i>
4	0.13 au (20 s)
	0.27 au (4 min.)
24	0.0 au (20 min.)
	0.0 au (4 min.)

17. The results in Table 3 demonstrate that the presence of oxygen in a composition of THPS, ammonium, chloride, and water renders the composition incapable of complexing iron (II).

18. The foams disclosed by Chartier contain 90 – 95 vol. % air, which is a much higher concentration of air than was present in the air-sparged test compositions described above. The presence of a greater concentration of air, and thus a higher concentration of oxygen, ensures that THPS, ammonium chloride, and water would undergo more rapid decomposition, *via* oxidation of TRIS, such as evidenced in Table 1 above.

19. Additionally, the foam of Chartier enforces a much higher surface area contact between air and the THPS, ammonium chloride, and water components than that achieved by simply sparging air through a solution of the same components. Greater surface area contact between the key reactants here –oxygen and TRIS – ensures greater rates and degrees of oxidation to TRIS oxide, which does not give rise to iron (II) complexation.

20. The foam that is explicitly recited in claim 1 of Chartier contains only 2 – 3 wt. % of THPS, whereas the control and test compositions contained 12 wt. % THPS. A lower concentration of THPS relative to the concentration of oxygen would ensure a more rapid and complete decomposition of a composition containing THPS, ammonium chloride, and water.

21. These features of the disclosed foam, viewed particularly in light of the results collected in Table 3, renders it a virtual certainty that the oxygenated foams of Chartier do not complex iron sulfide.

22. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application of any patent issuing thereon.

Respectfully submitted,

Date November 6, 2003 By Edward J. Valente  
Prof. Edward J. Valente

**APPENDIX A**  
***Curriculum Vitae***

**Edward Joseph Valente**

**Born:** January 16, 1949, in San Francisco, CA

**Education:** Ph.D. (Chemistry) University of Washington, Seattle; March, 1977

B.S. (Chemistry) University of California, Berkeley; March, 1971

**Positions:** Professor of Chemistry, Mississippi College, Clinton, MS; 1989-present.

Associate Professor of Chemistry, Mississippi College, Clinton, MS; 1983-1989.

Adjunct Professor of Chemistry, Jackson State University, Jackson, MS; 1988-present.

Visiting Senior Research Scientist, Smith, Kline & French R & D, King of Prussia, PA;  
Summer, 1988.

Post-doctoral Scholar, Department of Chemistry, University of Michigan, Ann Arbor, MI;  
1981-1983.

Instructor in Chemistry, Coastal Carolina Community College, Jacksonville, NC; 1979-1981.

Post-doctoral Research Associate; Department of Chemistry, University of North Carolina,  
Chapel Hill, NC; 1978-1979, Summer, 1980.

Post-doctoral Research Assistant; Department of Pharmaceutical Sciences, University of  
Southern California, Los Angeles, CA; 1977-1978.

**Professional Organizations:**

American Chemical Society (1976-present)

American Crystallographic Association (1980-present)

Mississippi Academy of Sciences (1983-present; Chair, Chemistry, 1989-91, 1998)

**Honors:** Chemist of the Year American Chemical Society, Mississippi Section, 1998-9

Distinguished Lecturer; School of Arts and Sciences, Mississippi College, 1998

Professor of the Year, Mississippi College, 2003-4

**Teaching:** Organic Chemistry, Medicinal Chemistry, General Chemistry, Instrumental Analysis

**Research Interests:**

Synthesis, spectroscopy, and structure of derivatives of 4-hydroxy-coumarin related to warfarin.

Nucleation in nozzle flow; electron diffraction studies of liquids and microcrystalline solids.

Structures of organic diastereomeric salts, aspects of molecular recognition.

Modulation of polar bond lengths and correlation with organic reactivity.

**Grants**

Dreyfus & Keck Foundations: A Modern Magnetic Resonance Spectroscopy for an Enhanced Chemistry Curriculum and Support of Local Two-Year College Organic Chemistry, 3/01-3/03.

National Science Foundation (CCLI -A&I), AModern Magnetic Resonance Spectroscopy for an Enhanced Chemistry Curriculum and Support of Local Two-Year College Organic Chemistry@, 3/01-3/03.

National Science Foundation (CCLI-A&I), Washington, DC; ASpectroscopy for Student-Designed General Chemistry Explorations and Advanced Chemistry, 9/99-8/01.

National Science Foundation (ILI), Washington, DC; ALuminescence, Scintillation and Spectrofluorimetry for Analytical and Bioanalytical Laboratories, 7/96-6/98.

Office of Naval Research, Bethesda, MD; Structure Property Relations (With J. D. Zubkowski, Jackson State University) 6/88-5/91;6/91-5/94; 6/94-5/97; renewal (6/97-5/99); renewal (6/99-5/01).

National Institutes of Health, Div. of Research Resources, Bethesda, MD, (AREA) "Xray Diffraction Support for Undergrad. Research"; 4/90 - 3/92.

National Science Foundation (ILI), Washington, DC; "Gas Chromatograph-Mass Spectrometry for a Modern Chemistry Curriculum"; 7/89 - 6/91.

National Science Foundation (ILI), Washington, DC; "Fourier-Transform Infrared Spectroscopy for a Modern Chemistry Curriculum"; 9/88 - 8/90.

American Heart Association, Mississippi Affiliate, Jackson, MS; "Anti-coagulants as Probes of Binding to Macromolecules", 7/84 - 6/87.

Research Corporation, Tucson, AZ; "Stability and Chemistry of Hexachloroarsenate (V) Salts", 6/84 - 6/85.

Southern Regional Education Board, Atlanta, GA; " Structure of Methyl Ketals of Warfarin and Related Compounds", 3/80.

**Attachment Publications**

Dissertation: "A Structural Study of Coumarin Anticoagulants and Other Derivatives of 4-Hydroxycoumarin", University of Washington, 1977. Advisors: E. C. Lingafelter (chemistry) & W. F. Trager (pharmaceutical sciences).

1. Crystal and Molecular Structure of Warfarin, a Clinical Oral Anticoagulant Drug. **E. J. Valente, W. F. Trager & L. H. Jensen.** Paper delivered before the American Crystallographic Association Meeting, March, 1974.
- 1a. Crystal and Molecular Structure and Absolute Configuration of (-)(S)-Warfarin. **E. J. Valente, W. F. Trager & L. H. Jensen,** *Acta Cryst.* **B31**, 954-960 (1975).
2. (-)-3-(1-Phenylpropyl)-4-hydroxycoumarin. **E. J. Valente, W. F. Trager & E. C. Lingafelter,** *Acta Cryst.* **B32**, 277-279 (1976).
3. The Structure of Warfarin in Solution. **E. J. Valente, E. C. Lingafelter, W. R. Porter & W. F. Trager,** *J. Med. Chem.* **20**, 1489-1493 (1977).
4. The Conformation of Selected 3-Substituted 4-Hydroxycoumarins in Solution by NMR: Warfarin and Phenprocoumon. **E. J. Valente, W. R. Porter & W. F. Trager,** *J. Med. Chem.* **21**, 231-234 (1978).
5. The Anomalous Chiroptical Properties of Warfarin and Phenprocoumon. **E. J. Valente & W. F. Trager,** *J. Med. Chem.* **21**, 141-143 (1978).

6. Conformation of Dihydropyran Rings: The Structures of Two  
3,4-Dihydro-2H,5H-pyrano[3,2-c][1]benzopyran-5-ones. **E. J. Valente**, B. D. Santarsiero & V.  
Schomaker, *J. Org. Chem.* **44**, 798-802 (1979).

7. Proton Magnetic Resonance Study of the In Vitro Decomposition of 4-Hydroxy-cyclophosphamide, a  
Microsomal Metabolite of Cyclophosphamide. E. J. Valente, K. K. Chan & K. L. Servis, Paper  
presented before the Academy of Pharmaceutical Sciences and Medicinal Chemistry, April, 1979.

7a. Proton Magnetic Resonance Studies of the Decomposition of 4-Hydroxycyclophos-phamide, a  
Microsomal Metabolite of Cyclophosphamide. **E. J. Valente**, K. K. Chan & K. L. Servis, *J. Pharm.  
Biomed. Sci.* **2**, 89-92 (1984).

8. Structure of a Gla-containing Dipeptide. The Crystal Structure of (D)-N-Carbobenzoxy-( $\chi$ ,  $\chi$ -ditertbutyl)- $\chi$ -carboxyglutamylglycine Ethyl Ester. **E. J. Valente**, R. G. Hiskey & D. J. Hodgson,  
*Biochem. Biophys. Acta* **379**, 466-468 (1979).

9. NMR Configuration Correlation of Amine Derivatives of  $\chi$ -Methyl- $\chi$ -methoxypenta-fluorophenylacetic  
Acid. L. R. Pohl, **E. J. Valente** & W. F. Trager, *J. Org. Chem.* **45**, 543-546 (1980).

10. The Synthesis of C-2 Isotopically Labelled Optically Pure Warfarin and Phen-procoumon. W. R.  
Porter, K. Kunze, **E. J. Valente** & W. F. Trager, *J. Labelled Compounds and Radiopharm.* **17**,  
763-773 (1980).

11. 2-Methyl-2-hydroxy-4-cyclohexyl-3,4-dihydro-2H,5H-pyrano[3,2-c][1]benzo-pyran-5-one. **E. J.  
Valente** & D. J. Hodgson, *Acta Cryst.* **B35**, 3099-3101 (1980).

12. L-Aspartylglycine. D. S. Eggleston, **E. J. Valente** & D. J. Hodgson, *Acta Cryst.* **B37**, 1431-1433 (1981).

13. L-Glutamylglycine. D. S. Eggleston, **E. J. Valente** & D. J. Hodgson, *Acta Cryst.* **B37**, 1433-1435 (1981).

14. Structure of Bis(4-methylpyridine)dichlorocopper(II): A Dislocated Linear Chain. Wayne E. Marsh, **E. J. Valente** & D. J. Hodgson, *Inorg. Chem. Acta*. **51**, 49-53 (1981).

15. Electron Diffraction Studies of Supersonic Jets. Paper before the IX Austin Symposium on Molecular Structure. Austin, TX. R. K. Heenan, **E. J. Valente** & L. S. Bartell, March, 1982

15a. Electron Diffraction Studies of Supersonic Jets, II: Formation of Benzene Clusters. R. K. Heenan, **E. J. Valente** & L. S. Bartell, *J. Chem. Phys.* **78**, 243-248 (1983).

16. Electron Diffraction Studies of Supersonic Jets, V: Low Temperature Crystalline Forms of SF<sub>6</sub>, SeF<sub>6</sub>, TeF<sub>6</sub>. **E. J. Valente** & L. S. Bartell, *J. Chem. Phys.* **79**, 2683-2685 (1983).

17. Electron Diffraction Studies of Supersonic Jets, VI: Microdrops of Benzene. **E. J. Valente** & L. S. Bartell, *J. Chem. Phys.* **80**, 1451-1457 (1984).

18. Electron Diffraction Studies of Supersonic Jets, VII: Liquid and Crystalline Carbon Tetrachloride. **E. J. Valente** & L. S. Bartell, *J. Chem. Phys.* **80**, 1458-1461 (1984).

19. Structure of Dihydropyran Rings. Paper before the American Chemical Society Meeting, Seattle, WA. **E. J. Valente**, L. S. Bartell & V. Schomaker, March, 1983.

19a. A Structure containing Diastereomers, (2S,2R)-trans- and (2R,4R)-cis-2-Hydroxy-2,4-dimethyl-3,4-dihydro-2H,5H-pyrano[3,2-c][1]benzopyran-5-one. **E. J. Valente** & V. Schomaker. *Acta Cryst. C40*, 1068-1070 (1984) and *Proc. Miss. Acad. Sci. (Suppl.) 29*, 14 (1984).

20. A Simple High-Yield Synthesis of 4,5-Dihydroxycoumarin. B. S. Castleberry & E. J. Valente. A paper presented at the 33rd SW/37th SE Regional Meeting of the American Chemical Society, Memphis TN, October, 1985.

20a. Open-cyclic Warfarin Isomerism: 5-Hydroxywarfarin. B.S. Castleberry, **E.J. Valente** & D. S. Eggleston, *J. Cryst. Spectr. Res.*, **20**(6), 583-93 (1990).

21. Electron Diffraction Studies of Supersonic Jets, 8: Nucleation of Various Phases of SF<sub>6</sub>, SeF<sub>6</sub>, and TeF<sub>6</sub>. L. S. Bartell, **E. J. Valente** & J. C. Caillat, *J. Phys. Chem.* **91**, 2498-2503 (1987).

22. Control Over the Structure of Clusters Generated in Supersonic Flow. L. S. Bartell, L. Harsanyi & **E. J. Valente**. A Paper before the International Symposium on the Physics and Chemistry of Small Clusters, Richmond, VA, October, 1986 and "Physics and Chemistry of Small Clusters", P. Jena, B. Rao, S. Khanna - Eds., NATO ASI Series B: Physics; vol. 158, pp 37-42, Plenum Press, NY 1987.

23. Structures of Five trans-2-Hydroxy and Methoxy-2-methyl-3,4-dihydro-4-aryl-2H,5H-pyrano[3,2-c][1]benzopyran-5-ones. **E. J. Valente**, D. S. Eggleston & V. Schomaker. *Acta Cryst. C42*, 1809-1813 (1986).

24. Structures of Four trans-2-Hydroxy and  
Methoxy-2-methyl-3,4-dihydro-4-alkyl-2H,5H-pyrano[3,2-c][1]benzopyran-5-ones. **E. J. Valente**, D.  
S. Eggleston & V. Schomaker, *Acta Cryst.* **C43**, 533-536 (1987).

25. Electron Diffraction Studies of Supersonic Jets. 11: Nucleation of Low-Temperature Phases of  
Perfluorocyclobutane. L. Harsanyi, L. S. Bartell & **E. J. Valente**, *J. Phys. Chem.* **92**, 4511-4514  
(1988).

26. Structures of cis-4-(2-Methoxyphenyl)- and  
trans-4-(3-Methoxy-4-hydroxyphenyl)-2-hydroxy-2-methyl-3,4-dihydro-2H,5H-pyrano[3,2-c][1]benz  
opyran-5-one. B. S. Castleberry, G. Ruggiero, **E. J. Valente** & D. S. Eggleston, *Acta Cryst.* **C44**,  
1281-1283 (1988).

27. Synthesis of  $^{13}\text{C}$ -Warfarin Labelled at the Hemiketal Carbon, and Its Resolution. V. H. Savell, Jr., **E.  
J. Valente** & D. S. Eggleston, *J. Labelled Comp. and Radiopharm.* **27**(6), 641-645 (1989).

28. ADiastereomeric Discrimination: Structural Aspects. @ **E. J. Valente**, G. Ruggiero, C. W. Miller, J.  
D. Zubkowski, and D. S. Eggleston. In Materials Synthesis and Characterization, Chapter 2, p 19 -  
60, Plenum Publishing Co., New York, NY (1997).

28a. Exploitation of Classical Resolution Patterns in Warfarin Analogs. E. J. Valente G. Ruggiero & D.  
S. Eggleston. A paper presented at the annual meeting of the American Crystallographic Association,  
Seattle, WA July, 1989. Abstract LA8.

29. Phases and Phase Changes of Molecular Clusters Generated in Supersonic Flow. L. S. Bartell, L. Harsanyi & E. J. Valente. *J. Phys. Chem.*, **93**, 6201 - 6205 (1989).

30. Structure of (−)-6-Methyl-6,12-methano-6H,12H,13H-[1]benzopyran [4,3-d][1,3]benzodioxocin-13-one. G. Ruggiero, E. J. Valente & D. S. Eggleston, *Acta Cryst.*, **C45**, 1182-1184 (1989).

31. Structures of (−)-cis-2-Hydroxy-4-(2-Phenylethyl) and cis-4-Phenyl-2-methyl-3,4-dihydro-2H,5H-pyrano[3,2-c][1]benzopyran-5-one. G. Ruggiero, E. J. Valente & D. S. Eggleston. *Acta Cryst.*, **C45**, 1369-1372 (1989).

32. Structures of (−)-trans-4-(3-Nitrophenyl) and (+)-trans-4-(4-Fluorophenyl)-2-Hydroxy-2-methyl-3,4-dihydro-2H,5H-pyrano[3,2-c][1]benzopyran-5-one. V. H. Savell, Jr., E. J. Valente & D. S. Eggleston, *Acta Cryst.*, **C45**, 1908-1911 (1989).

33. Structures of (−)-trans-4-(2,3-Dimethoxyphenyl) and (+)-trans-4-(4-Methyl-phenyl)-2-Hydroxy-2-methyl-3,4-dihydro-2H,5H-pyrano[3,2-c][1]benzopyran-5-one. G. Ruggiero, O. L. Platt, Jr., E. J. Valente & D. S. Eggleston, *Acta Cryst.* **C45**, 1740-1743 (1989).

34. Structure of (Phenyl)bis(4-hydroxybenzo-2H-pyran-2-one-3-yl)methane. E. J. Valente & D. S. Eggleston, *Acta Cryst.* **C45**, 785-787 (1989).

35. Test of an Empirical Criterion for Predicting Cluster Phases. L. S. Bartell, E. J. Valente & T. S. Dibble, *J. Phys. Chem.* **94**, 1452-1453 (1990).

36. Structural Variations in Dihydropyranyl Ketals: Aryl and Acyl Warfarin Derivatives. Gerard Ruggiero, Anson Lee Thaggard, **E. J. Valente** & D. S. Eggleston, *Acta Cryst.* **B46**, 629-637 (1990).

37. Dihydropyran Ring Conformations I: Structures of 2-Methoxy and Hydroxy 2,4-dimethyl-3,4-dihydro-2H,5H-pyrano[3,2-c][1]benzopyran-5-ones. **E. J. Valente** & D. S. Eggleston, *Acta Crystallographica* **B47**, 498-501 (1991).

38. Discrimination in Resolving Systems: Ephedrine -Mandelatic Acid. **E. J. Valente**, Jeff Zubkowski, and Drake S. Eggleston, *Chirality*, 4(8), 494-504, (1992).

38a. Structures of Diastereomeric Salts: 1R,2S-Epehedinium 2S-, (2S,2R), and 2R-Mandelite. **E. J. Valente**, Donald Thomas, Jimmy Boyd, Jeff Zubkowski, and Drake S. Eggleston, paper presented before the annual meeting of the American Crystallographic Association, Toledo OH, July, 1991. Abstract PI14.

38b. Patterns in Resolving Systems: Discrimination Between Ephedrine Salts of o-, m-, p-Halomandelates. Anthony Dribben, Steven Easley, Reid Bishop and **E. J. Valente**, a paper presented before the 56th Annual meeting of the Mississippi Academy of Sciences, Biloxi, MS, February, 1992.

39. Structure of (")-6,8-Dimethyl-6,12-methano-6H,12H,13H[1]benzopyran[4,3-d][1,3]benzodioxocin-13-one. **E. J. Valente**, Gerard Ruggiero, Drake S. Eggleston, *Acta Cryst.*, **C48**, 1635-1637 (1992).

40. Discrimination in Resolving Systems. II. Ephedrine - Substituted Mandelic Acids. **E. J. Valente**, C. W. Miller, J. D. Zubkowski, D. S. Eggleston and X. Shui. *Chirality* 7(8), 652-676 (1995).

40a. Discrimination in Resolving Systems: Ephedrine Halomandelates. E. J. Valente, Jeffrey Zubkowski, and Drake S. Eggleston. A paper presented before the annual meeting of the American Crystallographic Association, Pittsburgh PA, August, 1992.

40b. Structures and Properties of Three Ephedrinium Halomandelates. Chris Miller & **E. J. Valente** and Jeff Zubkowski. A paper presented before the 57th annual meeting of the Mississippi Academy of Sciences, Jackson MS, February, 1993.

40c. Structure-Property Correlations in Diastereomeric (-)-Ephedrinium Halomandelates. **E. J. Valente**, Chris Miller, Jeffrey Zubkowski, and Drake S. Eggleston. A paper presented before the annual meeting of the American Crystallographic Association, Albuquerque, NM, May, 1993.

40d. Correlation of Structure with Properties of Diastereomeric Ephedrinium Halomandelates. 58th Annual Mississippi Academy of Sciences, February, 1994.

40e. Correlation of Structure with Properties of Diastereomeric Ephedrinium Halomandelates. A paper presented before the annual meeting of the American Chemical Society, March, 1994.

41. Synthesis and Structure of Bis(1,4,7-trithiacyclodecane) ruthenium(II) Perchlorate: The Meso Diastereomer. Gregory J. Grant, Bruce M. McCosar, William N. Setzer, Jeffrey D. Zubkowski & **Edward J. Valente**. *Inorganica Chimica Acta* **244**, 73-77 (1996).

42. Epimerization of Cyanocobalamin. Improved Synthesis of Cyano-8-epicobalamin and Its Characterization by X-ray Crystallography, and  $^1\text{H}$ ,  $^{13}\text{C}$ , and  $^{15}\text{N}$  NMR. Kenneth L. Brown, Ziang Zou, Guo-Zhang Wu, Jeffrey D. Zubkowski, and **Edward J. Valente**. *Inorganic Chemistry*, **14**(12) 1621-1639 (1995).

43. The Synthesis and Structure of  $\text{W}_2\text{Cl}_4(\text{dppm})_2(\text{CH}_3\text{CN})$ . J. L. Eglin, E. Marie Hines, **E. J. Valente** and J. D. Zubkowski. *Inorganica Chimica Acta*, **229**(1-2), 113-119, (1995).

43a. The Synthesis of  $\text{W}_2\text{Cl}_4(\text{dmmp})_2(\text{CH}_3\text{CN})$ . Judith L. Eglin, E. Marie Hines, J. D. Zubkowski, and **E. J. Valente**. 26th ACS Southeastern Regional Conference of Undergraduate Student Chemists, Murfreesboro, TN, March 24-26, 1994.

44. 5-Hydroxycyclocoumarol: A Layer Disordered Non-centrosymmetric Phase. **E. J. Valente**. A paper presented at New Trends in Small Moiety Crystallography Symposium at the American Crystallographic Association Meeting, Atlanta, GA, June, 1994.

45. A Seven-coordinate Cobalt-EDTA Complex. Crystal and Molecular Structure of Aquo(ethylenediaminetriacetatoacetic acid)cobalt(III)dihydrate. D. L. Perry, J. D. Zubkowski, S. Lott, and **E. J. Valente**. *Inorganic Chemistry* (1995) **34**, 6409-6411. [See also Errata: *Inorg. Chem.* (1996)]

45a. Raman and Infrared Spectroscopy of the Cobalt-EDTA System. Eric. C. Faulques, Dale L. Perry, Jeffrey D. Zubkowski and **Edward J. Valente**. A paper presented at the Northeast Regional Meeting of the American Chemical Society, New York, NY, September, 1994.

46. Formation, Magnetic Properties and Structures of Copper (II) Levulinates. J. D. Zubkowski, D. Washington, N. Njorge, **E. J. Valente**, T. Cannon, C. D. Parks, P. Berdahl, D. Perry, *Polyhedron* **16**, 2341 - 2351 (1997).

46a. Synthesis and Structure of Copper (II) Levulinate. D'Angelo Washington, Novella Bridges, Tonya Cannon, C. Dean Parks, **E. J. Valente** and J. D. Zubkowski. A poster at the 1995 American Chemical Society (Mississippi Section) Mid-winter Symposium, Jackson, MS, January 20, 1995.

47. Copper(II) Nitrate Compounds with Heterocyclic Ligands: Structures of  $[\text{Cu}(\text{NO}_3)(2,2'\text{-dipyridyl})_2]\text{[NO}_3\text{]} \cdot \text{H}_2\text{O}$  and  $[\text{Cu}(\text{H}_2\text{O})(1,10\text{-phenanthroline})_2]\text{[NO}_3\text{]}_2$ . K. J. Catalan, S. Jackson, J. D. Zubkowski, D. L. Perry, **E. J. Valente**, L. A. Feliu and A. Polanco. *Polyhedron* **14**, 2165-2171 (1995).

47a. Copper(II) Nitrate Compounds with Heterocyclic Ligands: Structures of  $[\text{Cu}(\text{dipyridyl ketone hydrate})]\text{[NO}_3\text{]}_2 \cdot \text{H}_2\text{O}$ . C. Weathers, W. Durr, J. D. Zubkowski, **E. J. Valente** and D. L. Perry. A paper at the 209th American Chemical Society annual meeting, April 2-6, 1995 in Anaheim, CA; abstract 343t.

48. The Evaluation of Singlet-Triplet Separations for  $[\text{W}_2(\text{u-H}_2)(\text{u-Cl})\text{Cl}_4(\text{u-dmmp})_2]$  and  $[\text{W}_2(\text{u-H}_2)(\text{u-O}_2\text{CC}_6\text{H}_5)\text{Cl}_2(\text{P}(\text{C}_6\text{H}_5)_3)_2]$  Based on  $^{31}\text{P}$  NMR and Crystallographic Data. K. M. Carlson-Day, T. E. Concolino, J. L Eglin, C. Lin, T. Ren, **E. J. Valente** and J. D. Zubkowski. *Polyhedron* **15**, 4469 - 4478 (1996).

49. The Reactivity of  $\text{Re}_2\text{Cl}_6(\text{P}(\text{n-But})_3)_2$  with DPPE in the Presence of Nitriles. Laura T. Smith, Judith L. Eglin, **Edward J. Valente**, Jeffrey D. Zubkowski. A Paper at the SE/SW Regional ACS Meeting, Memphis, TN., 11/29-12/2/1995.

49a. Synthesis of a dinuclear tungsten complex,  $(\text{W}_2(\chi\text{-H})(\chi\text{-OC}_2\text{H}_5)(\chi\text{-}(\text{O}_2\text{CC}_6\text{H}_5)(\chi^1\text{-OC}_2\text{H}_5)\text{Cl}_2(\chi^2\text{-P,P=}-\text{dppp}))$  where dppp is 1,3-bis(diphenylphosphino)propane. K. M. Carlson-Day, J. L. Eglin, **E. J. Valente** & J. D. Zubkowski. *Inorganica Chimica Acta* **284**, 3000-3003 (1999).

50. Pseudoacids. I.: 4- And 5-Oxoacids. **E. J. Valente**, Jason F. Fuller & Jason D. Ball. *Acta Cryst.* **B54**, 162 - 173 (1998).

50a. Six-membered cyclic Pseudoacids: Synthesis and Structure of 2-carboxyphenyl acetaldehyde. Jason Fuller and **Edward Valente**, A Paper at the SE/SW Regional ACS Meeting, Memphis,TN., 11/29-12/2/1995.

50b. Open-cyclic Equilibrium in Levulinic Acid, Derivatives and Homologs. Jason D. Ball & **E. J. Valente**. A paper at the 59th Annual Meeting of the Mississippi Academy of Sciences, 2/19/95.

50c. Synthesis and Structure of Substituted Aryl Oxoacids. Jason Fuller, **E. J. Valente & J. D. Zubkowski**, A Paper at the 60th Annual Meeting of the Mississippi Academy of Sciences,Biloxi, MS, 2/22/96.

51. Heteronuclear NMR Studies of Cobalt Corrinoids. 18. Correlation of Structure and Magnetic Resonance Parameters in Base-On Cobalamins. K. L Brown, D. R. Evans, J. D. Zubkowski, and **E. J. Valente**. *Inorganic Chemistry* (1996), **35**, 415 - 423.

52. Facile generation of aziridines from the reaction of  $\gamma$ -diazoamides with tethered oximinoethers. M. C. McMills, D. L. Wright, J. D. Zubkowski, **E. J. Valente**. *Tetrahedron Letters* **37** (40), 7205 - 7208 (1996).

53. Synthesis and Characterizaton of a Novel Green Cobalt Corrinoid. K. L. Brown, S. Cheng, J. D. Zubkowski, and **E. J. Valente**. *Inorganic Chemistry*, **35**, 3442-3446 1996).

54. Structural and Spectroscopic Characterization of Ditungsten Halide Compounds. K.M Carlson-Day, J. L Eglin, C. Lin, T. Ren, **E. J. Valente**, and J. D. Zubkowski. *Inorganic Chemistry*, **35**(16), 4727-4732 (1996).

55. Quantum Chemical Conformational Analysis and X-Ray Structure of 4-Methyl-3-Thiosemicarbazide. C. C. Chambers, E. F. Archibong, S. M. Mazhari, A. Jabalameli, J. D. Zubkowski, R. H. Sullivan, **E. J. Valente**, C. J. Cramer and D. G. Truhlar, *Journal of Molecular Structure (Theoretical Chemistry)* **388**, 161-167 (1996); In Proceedings of the Fourth Conference on Current Trends in Computational Chemistry.

56. 4- and 5-nitroindane. Jason Fuller & **Edward J. Valente**. *Journal of Chemical Crystallography* **26**(12), 815 - 821 (1996).

57. Synthesis of a multiply-bonded ditungsten hydride complex,  $W_2(u\text{-H})(u\text{-O}_2CC_6H_5)Cl_4(dppm)_2$ . K. M. Carlson-Day, J. L. Eglin, **E. J. Valente** and J. D. Zubkowski. *Inorganica Chimica Acta* **244**, 151-154 (1996).

58. Cis Effects in the Cobalt Corrins. 1. The crystal structures of 10-chloroaquacobalamin perchlorate, 10-chlorocyanocobalamin and 10-chloromethylcobalamin. K. L. Brown, S. Cheng, X. Zou, J. D. Zubkowski, **E. J. Valente**, and H. Marks. *Inorganic Chemistry* **36**, 3666-3675 (1997).

59. The synthesis and characterization of  $\chi$ - $W_2Cl_4(dppp)_2$ . J. L. Eglin, **E.J. Valente**, Kristie R. Winfield & J. D. Zubkowski. *Inorganica Chimica Acta* **245**, 81-85 (1996).

60. Direct Detection by  $^{15}\text{N}$ -NMR of the Tryptophan Tryptophylquinone Aminoquinol Reaction Intermediate in Methylamine Dehydrogenase. G. R. Bishop, **E. J. Valente**, T. L. Whitehead, K. L.

Brown, R. T. Hicks, and V. L. Davidson. *Journal of the American Chemical Society* **118**, 12868-12869 (1996).

61. Study of Lead-Sulfur Compounds. The synthesis and Characterization of  $[\text{Pb}(\text{S}_2\text{CNEt}_2)_2(1,10\text{-phenanthroline})]_2$ . T. Hall, J. D. Zubkowski, **E. J. Valente**, D. L. Perry, L. A. Feliu and J. Garmon. *Journal of Chemical Crystallography* **27**(4), 251 - 255 (1997).
62. Study of coordination and ligand structure in cobalt-EDTA complexes with vibrational microspectroscopy. E. Faulques, D. L. Perry, S. Lott, J. D. Zubkowski, and **E. J. Valente**. *Spectrochimica Acta* **A54**, 869 - 878 (1998) .
63. Continuous Spectroscopic and Redox Tuning of Dinuclear Compounds: Chlorotetrakis( $\chi$ -N,N-diarylformamidinato)diruthenium(II,III). C. Lin, T. Ren, E. T. Smith, **E. J. Valente**, and J. D. Zubkowski. *Chemistry Letters* 753 - 754 (1997).
64. Side Chain Entropy and the Activation of Organocobalamins for Cobalt-Carbon Bond Homolysis: Synthesis, Characterization, and Thermolysis of the Neopentyl Derivative of a Unique Cobalamin Analog Lacking a c Side Chain. K. L. Brown, S. Cheng, J. D. Zubkowski and **E. J. Valente**, *Inorganic Chemistry* **36**(9), 1772 - 1781 (1997).
65.  $9\chi$ -Fluoro- $11\chi,17\chi,21$ -trihydroxy- $1,4$ -pregnadiene- $3,20$ -dione- $21$ -acetate (9-fluoro-prednisolone- $21$ -acetate). **Edward J. Valente**, Jeffrey D. Zubkowski, Ken S. Lee, A. J. Driesen, Kees Goubitz, Milco Numan, Hans Schenck, Henry J. Lee and Donghoon Ko. *Journal of Chemical Crystallography* **27**(4), 219 - 222 (1997).
66. The synthesis and characterization of  $\text{ReCl}_2(\text{dppe})_2$ ,  $\chi$ - $\text{Re}_2\text{Cl}_4(\text{dppe})_2$ . J. L. Eglin, L. T. Smith, **E. J. Valente** and J. D. Zubkowski. *Inorganica Chimica Acta* **268**, 151 - 157 (1998).

67. Structures of anti,Z-4,4-dimethyl-3-thiosemicbazide, syn,E,E-2,4-dimethyl-3-thiosemicbazide and syn,E,Z-1-cyclopentano-3-thiosemicbazone. **Valente, E. J., Zubkowski, J. D., Jabalameli, A., Mazhari, S. M., Venkatraman, S. & Sullivan, R. H.** *Journal of Chemical Crystallography* **28**(1), 27-33 (1998).

68. Pseudoacids. II.: 2-Acylbenzoic Acid Derivatives. **E. J. Valente, Samuel B. Martin & Larry D. Sullivan.** *Acta Crystallographica* **B54**, 264 - 276 (1998).

68a. Synthesis and Structure of Substituted Aryl Pseudoamides. **Samuel Martin & E. J. Valente.** A paper at the 61st Annual Meeting of the Mississippi Academy of Sciences, Biloxi, MS, 2/20/97.

68b. Synthesis of Some Nitroaryl Pseudoesters. **Samuel B. Martin & E. J. Valente.** 17<sup>th</sup> Undergraduate Research Conference, University of Memphis, Memphis, TN. 3/1/97.

69. W<sub>2</sub>(χ-H)<sub>2</sub>Cl<sub>4</sub>(χ-dppm)<sub>2</sub>: The Bulk Synthesis of a Bridging Hydride. **T. E. Concolino, J. L. Eglin, E. J. Valente & J. D. Zubkowski.** *Polyhedron* **16**(23), 4137 - 4141 (1997).

70. Discrimination in Resolving Systems. III. Pseudoephedrine - Mandelic Acid. **E. J. Valente, M. C. Moore, P. M. Knight.** *Chirality* **10**, 325 - 337 (1998).

70a. Discrimination in Resolving Systems: Pseudoephedrine - Mandelic Acid. **E. J. Valente, P. M. Knight, M. C. Moore.** A paper presented at the American Crystallographic Association Annual Meeting, St. Louis, MO; 7/24/97.

71. Synthesis, Spectroscopy and Electrochemistry of Tetrakis( $\chi$ -N,N-arylformamidinato) di(phenylacetylide) diruthenium(III). C. Lin, T. Ren, **E. J. Valente**, and J. D. Zubkowski. *J. Chem. Soc. Dalton Trans.* **1998**, 571 - 576.

72. Crystal structure of 2,2,4,7,7-pentamethyl-3,6-dithiaoctane tetracarbonyl tungsten (0). J. E. Cortes-Figuroa, J. D. Zubkowski & **E. J. Valente**. *J. Chem. Cryst.* **28**, 217 – 220 (1998).

73. Characterization of  $[\text{Re}_2\text{Cl}_3(\chi\text{-dppm})_2(\text{NCCH}_3)_2][\text{Cl}]$  and  $[\text{Re}_2\text{Cl}_3(\chi\text{-dppm})_2(\text{NCCH}_3)_2][\text{Cl}]\chi 2\text{CH}_2\text{Cl}_2$  by X-ray Crystallographic and Two-Dimensional NMR Spectroscopic Techniques. T. E. Concolino, J. L. Eglin, C. H. Hadden, R. P. Hicks, R. J. Staples, **E. J. Valente** & J. D. Zubkowski. *Journal of Cluster Science* **11**(4), 109-124 (2000).

74. 4- and 5-nitro-2-benzoylbenzoic acid. Samuel B. Martin and **Edward J. Valente**. *J. Chem. Cryst.* (1998) **28**, 203 - 207.

75.  $\text{Na}_2[(\chi\text{-EDTA-N,N=})(\chi\text{-O})(\chi\text{-S})\text{Mo}_2\text{O}_2]\chi 2\text{H}_2\text{O}$  **E. J. Valente**, J. D. Zubkowski and K. W. Hicks. *Acta Crystallographica C* **54**, 1265 - 1268 (1998).

76. Cyclodecanol. **E. J. Valente**, D. M. Pawar, E. A. Noe. *Acta Crystallographica C* **54**, 1302 - 1304 (1998).

77. Structural and Enzymatic Studies of a New Analog of Coenzyme B<sub>12</sub> with an  $\chi$ -Adenosyl Upper Axial Ligand. K. L. Brown, S. Cheng, X. Zou, G. Chen, **E. J. Valente**, J. D. Zubkowski and H. M. Marques. *Biochemistry* **37**, 9704 - 9715 (1998).

78. The Activation of Organocobalt Corrinoids for Carbon-Cobalt Bond Homolysis. K. L. Brown, D. R. Evans, S. Cheng, H. M. Marques, J. D. Zubkowski and **E. J. Valente**. Recent Research Developments in Inorganic Chemistry, Volume 1, Transworld Research Network, pp 1 - 11, 1998.

79. (Discrimination in Resolving Systems. IV.) Deoxyephedrine - Mandelic Acid Diastereomers. **E. J. Valente**, Marcus Harris, Kees Goubitz & Hans Schenk. *Zeitschrift für Kristallographie* **214**, 402 - 409, 1999.

80. syn,E-1-cyclopentano-4-ethyl-3-thiosemicarbazone and syn,E-1-cyclopentano-4-phenyl-3-thiosemicarbazone. Ramaiyer Venkatraman, Kristie Davis, Akilah Shelby, Jeffrey D. Zubkowski and **Edward J. Valente**. *J. Chem. Crystallogr.* **29**(4), 429 - 434, 1999.

81. Deconvoluting the Nature of Metal-axial-Alkynyl Interaction via Substituent Effects in Diruthenium-Phenylethyanyl Series; Linear Free Energy Relationships in Dinuclear Compounds VI, C. Lin, T. Ren, **E. J. Valente**, J. D. Zubkowski. *J. Organomet. Chem.* **579**, 114-121 (1999).

82. A convenient Preparation of a Bicyclo[3.3.3]-Undecane Derivative Containing Heteroatoms Through a Rearrangement Step. Tian, J.; Yan, Y.; Gong, B.; **Valente, E. J.**; Zubkowski, J. D. *Synth. Commun.* **28**(10), 1907 (1998).

83. Aqua(1,10-phenanthroline)(L-prolinato)copper (II) nitrate monohydrate. Ramaiyer Venkatraman, Jeffrey D. Zubkowski and Edward J. Valente. *Acta Crystallographica C* **55**, 1241 - 1243 (1999).

84. The Influence of Remote Substituents in Tetrakis( $\chi$ -N,N=-diarylformamidinato)dirhodium (II) compounds. Part 7. Linear Free Energy Relationships in Dinuclear Compounds. T. Ren, C. Lin, **E. J. Valente**, J. D. Zubkowski. *Inorg. Chim. Acta* **297**, 283 - 290 (2000).

85. Discrimination in Resolving Systems. V. Pseudoephedrine - Fluoromandelic Acid Diastereomers.

**Edward J. Valente**, Michael C. Moore. *Chirality* **12**, 16 - 25 (2000).

86. Inter-ring torsions in N-phenylmaleimide and its o-halo derivatives, an experimental and computational study. Christopher W. Miller, Charles E. Hoyle, **Edward J. Valente**, David H. Magers, E. Sonny Jönsson. *J. Phys. Chem. A* **103**(32), 6406-6412 (1999).

87. Synthesis and X-ray Crystal Structures of Mixed Sandwich Group 8 Cyclopentadienyl Complexes Containing Crown Trithioether Ligands. Gregory J. Grant, Terese Salupo-Bryant, Lisa A. Holt, Denise Y. Morrissey, Mary Jo Gray, Jeffrey D. Zubkowski, **Edward J. Valente**, Larry F. Mehne. *J. Organometallic Chem.* (1999) **587**, 207 - 214.

88. Synthesis, Characterization, and X-ray Crystallography of a Series of Ditungsten Complexes with Bis(diphenylphosphino)amine. J. D. Eglin, L. T. Smith, R. J. Staples, **E. J. Valente**, J. D. Zubkowski. *J. Organometallic Chem.* **596**, 136 - 143 (1999).

89. Kinetics of Substitution of Weakly Coordinating Nitrate by Chloride in  $(\chi^5\text{-Cp})\text{Ru}(\text{CO})\text{ER}_3\text{ONO}_2$  ( $\text{ER}_3 = \text{AsPh}_3, \text{PPh}_3, \text{P(p-anisyl)}_3, \text{PPh}_2(\text{o-anisyl}), \text{P(OPh)}_3$ ) in Dichloromethane. Cao, M., Do, L. V., Hoffman, N. W., Kwan, M-L., Little, J. K., McGilvray, J. M., Morris, C. B.; Soderberg, B. C., Wierzbicki, A., Cundari, T. R., Lake, C. M., **Valente, E. J.** *Organometallics* **20**, 2270-2279 (2001).

90. *cis*-tetracarbonylbis(tricyclohexylphosphine)molybdenum(0) and pentacarbonyl(tricyclohexylphosphine)molybdenum(0). Cortes-Figueroa, Jose, E., Velazquez, Madeline S., Ramos, Johanna, Jasinski, Jerry P., Keene, David A., Zubkowski, Jeffrey D., **Valente, Edward J.** *Acta Cryst. C* **56**, 1435 - 1437 (2000).

91. N-Arylmaleimide derivatives. Christopher W. Miller, Charles E. Hoyle, **Edward J. Valente**, Jeffrey D. Zubkowski, E. Sonny Jönsson. *J. Chem. Crystallogr.* **30** (9) 563-571 (2000).

92. Racemic and (+)-ethyl 2-acetamido-2-carboxy-5-oxohexanoate. Daniel A. Osborne, W. Reid Dreher, Jr., H. Ray Gore, Jr., Andrew Stoddard, and **Edward J. Valente**. *J. Chem. Crystallogr.* **30** (9) 583-588 (2000).

93. The crystal structure of bis-tricarbonylchromium complex of dibenzo[a,e]cyclooctatetraene. Cr(CO)<sub>3</sub>, orientation controlled by short C-H..O hydrogen bonding. M. Brown, J. D. Zubkowski, **E. J. Valente**, G.-Z. Yang, W. P. Henry. *Journal of Organometallic Chemistry* **613** 111-118 (2000).

94. Discrimination in Resolving Systems. VI. Comparison of the Diastereomers of Deoxyephedrinium and Ephedrinium 4-Fluoromandelates, **Edward J. Valente**, Tyler N. Smith and Marcus E. Harris. *Chirality* **13**, 244 - 250 (2001).

95. Heteroleptic Platinum (II) Complexes with Crown Thioether and Phosphine Ligands. I: The crystal structures of [Pt(9S3)(dppm)](PF<sub>6</sub>)<sub>2</sub> and [Pt(9S3)(P{phenyl}<sub>3</sub>)](PF<sub>6</sub>)<sub>2</sub>.2CH<sub>3</sub>NO<sub>2</sub>. Grant, G. J., Poullaos, I. M., Galas, D. F., VanDerveer, D. G., Zubkowski, J. D., **Valente**, E. J. *Inorganic Chemistry* **40**, 564 (2001).

96. Photopolymerization Characterization. Chris W. Miller, E. Sonny Jönsson, Charles E. Hoyle, Kalyanaraman Viswanathan, and **Edward J. Valente**. *Journal of Physical Chemistry* **105**, 2707 - 271 (2001).

97. Heteroleptic Platinum(II) Complexes of Macrocyclic Thioethers and Halides: Correlations Between <sup>195</sup>Pt NMR Chemical Shifts and Ligand Environment. Gregory J. Grant, Christopher G. Brandow,

David F. Galas, Jaime P. Davis, William T. Pennington, **Edward J. Valente**, Jeffrey D. Zubkowski. *J. Chem. Soc. Dalton Transactions* (2001) (submitted).

98. Heteroleptic Platinum(II) Complexes of Macroyclic Thioethers and Halides: The Crystal Structures of  $[\text{Pt}(9\text{S}3)\text{Cl}_2]$ ,  $[\text{Pt}(9\text{S}3)\text{Br}_2]$ , and  $[\text{Pt}(9\text{S}3)\text{I}_2]$  Gregory J. Grant, Christopher G. Brandow, David F. Galas, Jaime P. Davis, William T. Pennington, **Edward J. Valente**, Jeffrey D. Zubkowski, *Polyhedron* **20**, 3333-3342 (2001).

99. Unexpected thermal rearrangement of N-alkoxycarbonyl imidazole acrylazides to imidazo[1,5-c]pyrimidinone or imidazo[4,5-c]pyridinone. Hongtao Yu, **Edward Valente** *Tetrahedron Letters* **44**(33), 5879 - 5881 (2002).

100. UV-VIS Spectroscopy of 2,4,6-Trinitrotoluene-Hydroxide Reaction. Deborah R. Felt, Steven L. Larson, and **Edward J. Valente**. *Chemosphere* **49**, 287-295 (2002). And, the same has appeared as a technical report as document ERDC/EL TR-02-22, August, 2002.

101. Probing the nature of the Co(III) ion in the cobalamins. Spectroscopic and structural investigations of the reactions of aquacobalamin (vitamin B12a) with ambident nucleophiles. C.B. Perry, M. A. Fernandes, K.L. Brown, X. Zou, E. J. Valente, and H. M. Marques. *Eur. J. Inorg. Chem.* **136**, 2095-2107 (2003).

102. Pseudoacids. III. Formation and structures of new cyclic oxocarboxylic acids. W. John Cooper, Tyler N. Smith, Andrea K. Barker, Jason A. Webb, Edward J. Valente. *Journal of Chemical Crystallography* **33** (5-6), 373-382 (2003).

103. Meisenheimer complex from picric acid and diisopropylcarbodiimide. Rabih O. Al-Kaysi, David Creed, **Edward J. Valente**, *Journal of Chemical Crystallography* (submitted) (2003).